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FUZE IMMOBILIZATION WITH EXPLOSIVE-COMPATIBLE POLYMERS.(U)
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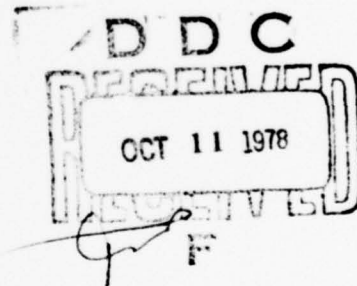
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RESEARCH AND TECHNOLOGY DEPARTMENT

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER 14 NSWC/WOL TR-78-55	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER (Kept)
4. TITLE (and Subtitle) Fuze Immobilization with Explosive- Compatible Polymers		5. TYPE OF REPORT & PERIOD COVERED Final - 15 April 1977 - 30 May 1977
7. AUTHOR(s) 10 Harry Heller		6. PERFORMING ORG. REPORT NUMBER
9. PERFORMING ORGANIZATION NAME AND ADDRESS Naval Surface Weapons Center White Oak, Silver Spring, Maryland 20910		8. CONTRACT OR GRANT NUMBER(s)
11. CONTROLLING OFFICE NAME AND ADDRESS 1234 P.		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 62734N;SF34-373; ZF34-373-001;CR-12-BA;
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) 16 F34373		12. REPORT DATE 11 14 July 1978
		13. NUMBER OF PAGES 28
		15. SECURITY CLASS. (of this report) UNCLASSIFIED
		15a. DECLASSIFICATION DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited 17 ZF34373 001		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Explosive Chemical Reactivity Fuze Train Explosives Explosive-Compatible Polymers		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A paper study was made of several classes of thermosetting polymer systems and their chemical compatibility with Navy fuze train explosives.		

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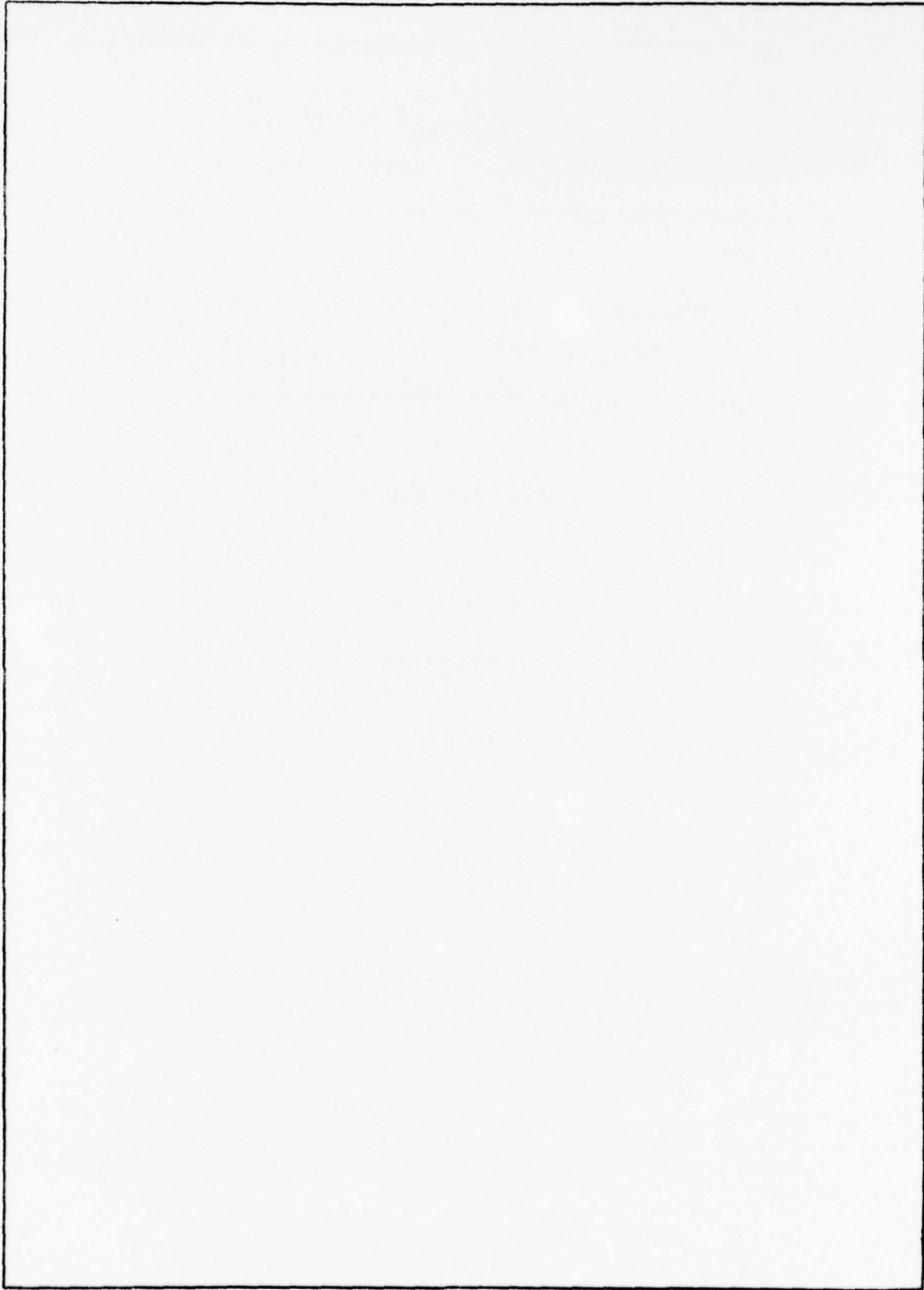
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SUMMARY

This is a final report on a study made for the Naval Explosive Ordnance Disposal Facility, Indian Head (NEODF/IH), Maryland.

A limited paper study was made of the probable compatibility of several classes of thermosetting polymer systems with Navy fuze train and booster explosives. The objective was to establish a basis for future development of a polymer system which could be used to immobilize the mechanical components of fuze trains in weapons slated for destruction by explosive ordnance disposal personnel.

The author wishes to acknowledge the assistance of Dr. Lyle Malotky of NEODF/IH. The listing of commercial materials implies no criticism or endorsement by the Naval Surface Weapons Center.

Julius W. Enig
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1. INTRODUCTION

1.1 In recent years there has been a growing requirement for improved thermosetting polymers for use as liners, sealants, adhesives and potting compounds in conjunction with explosives in advanced weapon systems. Thermosetting polymers are also being used as binders for a relatively new class of cast-cured military explosives called plastic bonded explosives (PBX).

1.2 It is necessary that the polymer composition and the explosive be chemically compatible in those instances where physical contact between the two materials is possible. This report is a limited paper study of several classes of thermosetting polymer systems and their probable compatibility with Navy fuze train and booster explosives. The objective was to establish a basis for future development of a polymer system for use in contact with explosives superior to the one currently in use.

2. COMPOSITIONAL REQUIREMENTS

2.1 For successful application under field conditions, a useful polymer composition must have many more desirable properties in addition to explosive compatibility. An ideal list of properties is shown below. However, on a more practical level, a single composition with all of these properties is probably unattainable.

- a. Compatible with all the explosive components of Navy fuze trains.
- b. Cures to form an adhesive, non-brittle, rigid, void-filling solid.
- c. Cures to a Shore A2 hardness greater than 50 in air or water in 15 minutes (a gel time of three minutes is desirable).
- d. Cure rate modifiable so as to function from -20 to 60°C or, if that is not possible, from -10 to 50°C.
- e. The system components must be easy to handle and be without excessive hazard to the operator.

f. The maximum viscosity of the polymer system at 25°C should be about 5000 cp. At minimum use temperature, the maximum viscosity is to be 50,000 cp.

3. EXPLOSIVE TRAINS

3.1 An explosive train is an assembly of combustible and explosive elements arranged in order of decreasing sensitivity inside a fuze. The function of the explosive train is to accomplish the controlled augmentation of a small impulse into one of sufficient energy to cause the main charge of the weapon to function reliably. These elements may include primers, detonators, relays, delays, leads and boosters.

3.2 Safety is a basic consideration throughout the life cycle of every weapon. An important aspect of system safety is that of protecting against initiation of booster or main charge explosive by stimuli incidental to normal handling, shipping, storage or launching and accidents that may occur during these operations. If an explosive-filled weapon fails to function as intended the safing mechanisms built into the explosive train may no longer be operating as designed. It is safer to jam or immobilize the mechanical/explosive components of the weapon to prevent movement during transportation and handling of the fuze weapon. This can be accomplished by injecting a liquid, thermosetting polymer. While the explosive elements in a train are normally sealed in containers, these may be ruptured in an accident. Therefore, any thermosetting resin used to immobilize the fuze train should be compatible with any explosive compound it can accidentally contact.

4. EXPLOSIVE TRAIN MATERIALS

4.1 Explosives currently approved for Navy usage* in primers, detonators, relays, leads and boosters are listed in Table 1. Given the large number of diverse energetic materials that may be present in an explosive train, finding a single thermosetting polymer that is compatible with all of these materials, and in addition has the required cure characteristics, poses a formidable task.

4.2 In an attempt to simplify this task, the explosive ingredients in Table 1 have been grouped according to similar chemical structure and listed in Table 2. Those ingredients which probably are compatible with all or most polymers were left out of Table 2. They include such materials as graphite, wax, polyisobutylene, etc. The listing is somewhat arbitrary since these explosives do not always fit into neat categories.

*Does not include delays and other pyrotechnics.

TABLE 1 - NAVY FUZE TRAIN AND BOOSTER EXPLOSIVES

<u>Principal Use</u>	<u>Explosive</u>	<u>Composition</u>	<u>Weight Percent</u>
Primer	AN #6 Primer Mix	Potassium Chlorate Antimony Sulfide Lead Azide Carborundum	33.4 33.3 28.3 5.0
Primer	DDNP	Diazodinitrophenol (DDNP)	100.0
Primer, Detonator	DDNP /KClO ₃	DDNP Potassium Chlorate	75.0 25.0
Primer	DDNP/KClO ₃ /Nitrostarch	DDNP/KClO ₃ (75/25) milled in a 2.4% solution of Nitrostarch	---
Primer	F.A. 70 Mix	Potassium Chlorate Antimony Sulfide Lead Thiocyanate TNT	53.0 17.0 25.0 5.0
Primer	F.A. 90A Mix	Lead Thiocyanate Antimony Sulfide Potassium Chlorate PETN	25.0 12.0 53.0 10.0
Primers, Detonators	Lead Azide	Lead Azide	100.0
Primers, Detonators	Lead Styphnate	Lead Styphnate	100.0

TABLE 1 - NAVY FUZE TRAIN AND BOOSTER EXPLOSIVES (Cont'd)

<u>Principal Use</u>	<u>Explosive</u>	<u>Composition</u>	<u>Weight Percent</u>
Primer	NOL #60 Primer Mix	Antimony Sulfide	10.0
		Barium Nitrate	25.0
		Lead Styphnate Basic	60.0
		Tetracene	5.0
Detonators, Primers	NOL No. 130 Primer Mix	Antimony Sulfide	15.0
		Barium Nitrate	20.0
		Lead Azide	20.0
		Lead Styphnate, Basic	40.0
		Tetracene	5.0
Primer	P.A. No. 100 Primer Mix	Potassium Chlorate	53.0
		Antimony Sulfide	17.0
		Lead Thiocyanate	25.0
		Lead Azide	5.0
Primer	P.A. No. 101 Primer Mix	Lead Styphnate, Basic	53.0
		Antimony Sulfide	10.0
		Barium Nitrate	22.0
		Aluminum Powder	10.0
		Tetracene	5.0
Detonator	PETN	Pentaerythritol Tetranitrate	100.0
Boosters, Leads	CH-6	RDX	97.5
		Calcium Stearate	1.5
		Graphite	0.5
		Polyisobutylene	0.5
Lead, Booster	DIPAM	Diaminohexanitrobiphenyl	100.0

TABLE 1 - NAVY FUZE TRAIN AND BOOSTER EXPLOSIVES (Cont'd)

<u>Principal Use</u>	<u>Explosive</u>	<u>Composition</u>	<u>Weight Percent</u>
Delay	F.A. 878 Mix	Barium Nitrate Lead Peroxide Zirconium, Granular Zirconium Powdered PETN	20.0 20.0 32.5 7.5 20.0
Lead, Booster	HNS	Hexanitrostilbene	100.0
Booster	PBXN-5	HMX Copolymer (Viton A)	95.0 5.0
Booster	PBXN-6	RDX Copolymer (Viton A)	95.0 5.0
Booster	Comp A-3	RDX Wax	91.0 9.0
Booster	Tetryl*	Trinitrophenylmethyl Nitramine	100.0
Booster	Comp A-5**	RDX Stearic Acid	99.0 1.0

*In process of being phased out

**Nonservice approved but allowed under restricted conditions

TABLE 2 - EXPLOSIVES GROUPED ACCORDING TO STRUCTURE

<u>Structure</u>	<u>Explosive</u>
1. <u>Aromatic C-Nitro Compounds</u>	TNT DIPAM HNS
2. <u>Aminoguanidine Derivative</u>	Tetracene
3. <u>Chlorates</u>	Potassium Chlorate
4. <u>Diazo Compounds</u>	Lead Azide DDNP
5. <u>Nitrophenol Salt</u>	Lead Styphnate
6. <u>Nitramines</u>	RDX HMX Tetryl
7. <u>Nitrate Ester</u>	PETN

5. CHEMICAL REACTIVITY OF FUZE TRAIN EXPLOSIVES5.1 Aromatic C-Nitro Compounds5.1.1 TNT: 2,4,6-Trinitrotoluene

5.1.1.1 TNT is relatively nonreactive, but it can form compounds by either association or reaction. With basic compounds such as aniline, pyridine, diphenylamine, the naphthylamines, the toluidines and carbazole, it forms molecular compounds having characteristic colors and melting points. Alkalis, alkoxides and ammonia react with TNT to form dangerously sensitive compounds. A mixture of TNT and solid potassium hydroxide bursts into flame when heated to about 80°C. Sodium carbonate reacts with TNT to form a black solid, which melts above 200°C with decomposition and is as sensitive to impact and heat as tetryl. Sodium sulfide decomposes TNT with the formation of nonexplosive products.

5.1.1.2 Exposure to sunlight or ultraviolet light in the presence of oxygen causes progressive discoloration and decomposition with increased sensitivity to impact. At 50°C, ethanol reacts with TNT. The products formed include ethylnitrite. TNT reacts to some extent with iron oxide as indicated by a decrease in high temperature stability. Potassium nitrite is incompatible with TNT. TNT is compatible with nitrates, potassium chlorate and black powder.

5.1.1.3 The methyl group in TNT is strongly activated by the nitro groups present in TNT. This explains why TNT readily reacts with benzaldehyde, formaldehyde and other aldehydes in alkaline media. These reactions are exothermic and may become violent enough for the mixture to ignite. The methyl group of TNT is readily oxidizable, forming trinitrobenzoic acid which in turn loses CO₂ to form trinitrobenzene.

5.1.1.4 TNT, like other highly nitrated aromatic hydrocarbons, easily forms addition products with polycyclic hydrocarbons and amines. Aromatic nitro compounds react with free radicals which explain their inhibiting effect on addition polymerization of vinyl compounds. Reference 1 reviews the reactions of polynitro aromatic compounds with bases.

5.1.2 DIPAM: 3,3'-Diamino-2,2',4,4',6,6'-hexanitrobiphenyl and HNS: 2,2',4,4',6,6'-Hexanitrostilbene

5.1.2.1 The reactivity of both DIPAM and HNS with alkalis and bases should be about the same as TNT. Neither explosive contains

1. Baliah, V. and Ramakrishnan, V., J. Annamalai Univ. Pt. B23 59 (1961)

an activated methyl group as in TNT. Although DIPAM contains two amino groups, the electron withdrawing effect of the nitro groups causes the compound to be somewhat acidic.

5.2 Aminoquanidine Derivatives

5.2.1 Tetracene: 4-Guanyl-1-(nitrosoaminoquanyl)-1-tetrazene

5.2.1.1 Boiling water decomposes tetracene with the liberation of nitrogen. It is hydrolyzed by sodium hydroxide yielding ammonia and cyanamide among the products. Tetracene reacts with an excess of silver nitrate to form a double salt and forms explosive salts such as the perchlorate. Tetracene has basic properties and is soluble in concentrated hydrochloric acid.

5.3 Chlorates

5.3.1 Potassium Chlorate: KClO_3

5.3.1.1 Pure KClO_3 has no explosive properties but when used in admixture with fuels such as sugar, starch or aluminum powder it forms an explosive highly sensitive to mechanical action, especially to friction. It is known that ammonium salts (e.g. ammonium nitrate) must not be added to chlorate compositions since a double exchange reaction may occur resulting in the formation of ammonium chlorate, an unstable compound which decomposes spontaneously.

5.4 Diazo Compounds

5.4.1 DDNP: 4,5-Dinitrobenzene-2-diazo-1-oxide; Diazodinitrophenol

5.4.1.1 A dilute solution of sodium hydroxide decomposes DDNP with the liberation of nitrogen. DDNP is not decomposed by concentrated acids at room temperature.

5.4.2 Lead Azide: $\text{Pb}(\text{N}_3)_2$

5.4.2.1 Lead azide, like hydrazoic acid, can undergo oxidation and reduction reactions. Lead azide is decomposed by a 10 percent solution of sodium hydroxide with the formation of lead hydroxide and sodium azide. When dissolved in aqueous ammonium acetate, lead azide reacts with potassium dichromate to form potassium azide and lead chromate. Lead azide is insoluble in an aqueous solution of ammonia, but acetic acid causes it to decompose. It is partially decomposed by atmospheric oxygen to form free hydrazoic acid, nitrogen and ammonia. This reaction is promoted by the presence of carbon dioxide in the air. Lead azide is completely decomposed by the action of dilute nitric or acetic acid in which sodium nitrite has been dissolved.

5.4.2.2 When boiled in water, lead azide undergoes slow decomposition with the evolution of hydrazoic acid. In aqueous suspension, lead azide is oxidized by ceric sulfate. Lead azide turns yellow when exposed to light. Dry lead azide does not react with or corrode the common metals. However, in the presence of moisture it corrodes many, if not most metals; and with copper forms the extremely sensitive and dangerous copper azide.

5.5 Nitramines

5.5.1 RDX: Cyclotrimethylenetrinitramine; Hexahydro-1,3,5-trinitro-S-triazine

5.5.1.1 RDX is hydrolyzed slowly when treated with boiling, dilute sulfuric acid or sodium hydroxide to yield ammonia, formaldehyde and nitric acid. RDX dissolved in cold concentrated sulfuric acid decomposes when the solution is allowed to stand. It is reduced when dissolved in hot phenol, yielding methylamine, nitrous acid, and hydrocyanic acid. If RDX is suspended in ethanol and reduced, both hydrolysis and reduction take place and the products are methylamine, nitrous acid, ammonia and formaldehyde. With diphenylamine and sulfuric acid, RDX gives a strong blue color. Light has a negligible effect on the stability of RDX. RDX dissolves in and forms eutectics in molten, nitrated aromatic hydrocarbons, substituted urea derivatives, and camphor.

5.5.2 HMX: Cyclotetramethylenetetranitramine; 1,3,5,7-tetranitro-1,3,5,7-tetrazacyclooctane

5.5.2.1 The chemical reactivity of HMX is similar to that of RDX. HMX is more resistant to decomposition by sodium hydroxide than RDX.

5.5.3 Tetryl: 2,4,6-Trinitrophenylmethylnitramine

5.5.3.1 Tetryl reacts with aqueous alkali to yield metallic picrates, nitrites and methylamine. When tetryl is boiled in a solution of sodium carbonate or potassium or sodium hydroxide, the nitramino group is hydrolyzed. The products are picric acid, methylamine and nitrous acid. When heated with alcoholic ammonia, a picramide is formed. Tetryl reacts with aniline in benzene solution even at room temperature to form 2,4,6-trinitrodiphenylamine and methylnitramine. When heated with phenol, tetryl is converted into trinitrophenylmethylamine. With tin and hydrochloric acid, tetryl is reduced and hydrolyzed to form 2,4,6-triaminophenol. Tetryl combines at room temperature with an excess of sodium sulfide in which the nitro groups are reduced with the formation of water soluble, nonexplosive products. Aqueous sodium sulfite decomposes tetryl into nonexplosive products but the reaction is slow unless the solution is heated to 80-90°C.

5.5.3.2 Although boiling with dilute sulfuric acid has no effect, if a solution of tetryl in cold, concentrated sulfuric acid is allowed to stand, nitric acid and 2,4,6-trinitrophenylmethanamine or N-methyl picramide are formed. If this reaction is carried out in the presence of mercury, the nitric acid is reduced to nitric oxide. Tetryl and TNT form an addition compound in the ratio of one mole of tetryl and two moles of TNT, which readily dissociates and melts at about 60°C. Like many explosives, the decomposition rate of tetryl increases sharply (~50 times) at its melting point. If tetryl contains other compounds which lower its melting point, the mixture begins to decompose at a lower temperature corresponding to the melting point of the mixture.

5.6 Nitrate Esters

5.6.1 PETN: Pentaerythritol Tetranitrate

5.6.1.1 Due to its symmetrical structure, PETN is highly resistant to many reagents. Thus PETN, differing from most nitric esters, is not readily decomposed by sodium sulfide at 50°C. But it is decomposed rapidly by boiling in a ferrous chloride solution. Boiling in a dilute solution of sodium hydroxide causes very slow decomposition.

5.6.1.2 At 125°C, under pressure, hydrolysis proceeds quite rapidly, and is accelerated by the presence of 0.1 percent of nitric acid. Dilute sodium hydroxide solution causes PETN to hydrolyze more rapidly than acidified water. PETN neither reduces Fehling's reagent nor enters into addition products with any aromatic nitro compounds. The addition to PETN of TNT and other nitro compounds decreases its stability at elevated temperatures (120-135°C). Reference 2 discusses a number of materials which are incompatible with PETN.

5.7 Nitrophenol Salts

5.7.1 Lead Styphnate: Lead 2,4,6-trinitroresorcinate

5.7.1.1 Lead styphnate is decomposed by concentrated nitric or sulfuric acid. When dissolved in ammonium acetate solution, it reacts with potassium dichromate to form potassium styphnate and lead chromate. Neutral lead styphnate reacts at 50°C with pyridine to form an addition product.

5.8 Pyrotechnic Compositions

5.8.1 The critical nature of the hygroscopicity of pyrotechnic compositions is due not only to the adverse effect of the adsorption of an inert diluent on the burning rate but also

2. Rogers, R. N. and Morris, E. D. Jr., Anal. Chem. 38, 412 (1966)

to the effect of moisture in promoting chemical instability. Powdered aluminum undergoes reaction with moisture to yield aluminum hydroxide and hydrogen gas and absorbed moisture may induce the reaction of other ingredients with this metal, its reaction products, or each other. Barium nitrate can react in the presence of moisture with metallic fuels. The hydrogen produced by the above reactions can reduce nitrates with the formation of ammonia which in turn can react with many explosive compounds. Zirconium combines high stability with low ignition and initiation temperatures. Very finely divided zirconium is exceedingly sensitive to accidental ignition by static electricity. Coarse zirconium powder is quite stable in the presence of air and/or water.

6. COMPATIBILITY OF VARIOUS MATERIALS WITH EXPLOSIVES

6.1 When normally inert materials are mixed with explosives, the properties of the resulting mixture cannot be predicted from the separate properties of the components. The components of the mixture may be "incompatible" with each other so that the system will not perform its intended function for one reason or another. As normally used in explosive parlance "incompatibility" is not a precise term and may refer to one or more different phenomena (Reference 3):

a. A chemical reaction between the additive and the components of the explosive which gives rise to a significant increase or decrease in sensitivity or decrease in thermal stability or the chemical destruction of one or more components of the explosive.

b. Migration of an explosive component into an adjoining substrate leading to changes in mechanical, physical, or explosive properties in either the explosive or substrate.

c. The formation of a low melting eutectic which may exude from the weapon during high temperature storage.

This report will only consider compatibilities of the type defined in (a) above.

7. TEST METHODS FOR EVALUATING COMPATIBILITY OF MATERIALS WITH EXPLOSIVES

7.1 Thermal and chemical decomposition kinetics of most explosives are extremely complex, and their reaction mechanisms are not clearly understood. Therefore, the most common analytical techniques for determining chemical and thermal explosive compatibilities are largely based on empirical methods. A major weakness of these tests is that they provide no indication of the type of reaction occurring or of the reaction products formed. It is often

3. Rogers, R. N., Ind Eng. Chem., Products Res. and Dev. 1, 169 (1962)

difficult to predict the relative compatibility of explosive mixtures over long time periods at lower temperatures, based on the data obtained from accelerated tests at higher temperatures. Scaling effects are usually important but are difficult to predict from small scale tests.

7.2 A large number of techniques and test procedures have been used to detect instability of explosives in contact with other materials. Some of the more common ones are given below.

7.2.1 Differential thermal analysis (DTA) - One of the more commonly used techniques in stability testing. The temperature of the explosive sample is measured and compared to a thermally-inert reference material heated and cooled under the same conditions. The DTA method is used to detect thermal changes or transitions. The usual changes observed are chemical reactions and phase transitions such as solid 1 to solid 2, solid to liquid, boiling, vaporization, fusion and glass transitions in polymers (References 4 and 5).

7.2.2 Differential scanning calorimetry (DSC) - Overcomes the disadvantages of conventional DTA equipment. Although sample and reference material are subjected to increasing or decreasing temperatures as in DTA, an additional control system detects any difference between sample and reference temperature and changes heat applied to maintain both at the same temperature. DSC measures the difference in electrical power required to maintain sample and reference at desired temperature and provides information on changes in chemical state and exothermic or endothermic chemical changes (Reference 6).

7.2.3 Thermal gravimetric analysis (TGA) - Measures the weight loss in a sample heated at elevated temperatures (Reference 7).

7.2.4 Mass spectrometer - An instrument for separating and determining the relative abundance of ions of different mass

4. Barefoot, R. D. and Whitman, C. L., "Compatibility of Nitrate and Nitrate Esters," Symposium on the Compatibility of Plastics and Other Materials with Explosives, Propellants, and Pyrotechnics, American Defense Preparedness Association, 27-29 Apr 1976
5. McKown, G. L., Pankow, J. F., and Lasseigne, A. H., "Effects of Copper and Heavy Metals on the Sensitivity of Pyrotechnic Materials," ibid
6. Fossum, J. H. and Weu, W. Y., "A Comparison of the Analytical Techniques for Testing the Compatibility of Polymers with High Energy Materials," ibid
7. Gould, D. J., et. al., "The Compatibility of PBX-9404 and Delrin," ibid

to charge ratio produced by bombarding the sample with electrons. It has the distinct advantage of identifying reaction products and characterizing starting materials and thus provides more useful information than more empirical methods of incompatibility testing (Reference 6).

7.2.5 Gas chromatography - Allows for the analysis of the gases evolved after accelerated thermal aging of an explosive sample (References 6, 8-14). Should volatile decomposition products be formed, they can be separated, identified and measured with the gas chromatograph. The use of the mass spectrometer in conjunction with the gas chromatograph is highly recommended for compatibility testing.

7.2.6 Chemiluminescence - If nitrogen oxides are the main products of decomposition during compatibility testing, and they usually are, this method is ideal for routine testing. Whereas 48 hours are required for the vacuum stability test, the chemiluminescence test requires one or two hours (Reference 6).

7.2.7 Colorimetry and Spectrophotometry - Uses the Griess-Saltzman reaction for the detection and measurement of nitrogen oxides. Main advantage is that special instrumentation is not required since most laboratories have some type of colorimeter or spectrophotometer. Preparation of special reagents is the main disadvantage (Reference 6).

7.2.8 Wet chemical methods - Very limited use. Can be used to convert compounds with limited volatility to more volatile ones for tests by gas chromatography and mass spectrometry.

7.2.9 Thin layer chromatography (TLC) - A micro type of chromatography in which a thin layer, usually silica gel spread on glass plates or plastic film, is the adsorbent. Drops of test solutions are placed along one edge of a coated plate, and this edge then dipped into a solvent. The solvent carries the constituents of the test solution up the thin layer in a selective separation, so that various identifying tests can be carried out. Both qualitative and quantitative analyses of explosives can be carried out by TLC.

8. Anal. Chem., 39 (11), 1315 (1967)
9. Anal. Chem., 36 (12), 2301 (1964)
10. Mem. Poud., 1964, p. 46
11. Mem. Poud., 1965, p. 164
12. J. Ind. Expl. Soc. Japan, 28, 146 (Mar 1967)
13. J. Chromat., 31, 551 (1967)
14. "Safety and Performance Tests for Qualification of Explosives," NAVORD OD44811, Vol. 1, Naval Weapons Center, China Lake, California, 1 Jan 1972

Purity and amount of thermal degradation of explosives have been assessed by this method. The compatibility of explosive mixtures, after some accelerated heating cycle, can also be determined. This method is especially useful if solid decomposition products are formed (References 15-24).

7.2.10 The Vacuum Stability/Compatibility Test

7.2.10.1 The test sample is heated in an evacuated system and the volume of evolved gases is measured over a standard time period and temperature. To determine compatibility of an explosive with an additive, separate runs are made with the additive, the explosive and the mixture. A chemical reaction is indicated if the gas volume of the mixture is greater than the sum of the gas volumes of the inert and explosive samples.

7.2.10.2 The vacuum stability/compatibility test has not been standardized. Temperatures of 90°, 100° and 120°C, time periods of 40 and 48 hours, and different sample weights are used by different laboratories. The Navy has standardized both the vacuum stability and explosive compatibility tests. The standard tests will appear in the revision of Reference 14. A complete description of the vacuum stability apparatus and the procedure used by the Naval Surface Weapons Center is given in Reference 25. Similar tests for stability and compatibility are given in References 26 and 27.

7.2.10.3 The vacuum stability and compatibility tests have a number of shortcomings. The method is empirical and based on unvalidated assumptions that incompatibility will result in the formation of noncondensable gases. For a given system this may or may not be true. The test provides only fair reproducibility, especially from laboratory to laboratory. It is slow (48 hours)

15. J. Chromatog., 24, 236 (1966)
16. Ibid., 31, 120, 551, 606 (1967)
17. Ibid., 38, 508 (1968)
18. Ibid., 51, 243 (1970)
19. Ibid., 62, 417 (1971)
20. Explosivestoffe, Sep 1966, p. 193
21. Ibid., Feb 1967, p. 25
22. Ibid., Jan 1971
23. Anal. Chem., 36 (12), 2301 (1964)
24. Nature, 216, 1168 (1967)
25. Simmons, H. T. Jr., "The Vacuum Thermal Stability Test for Explosives," NOLTR 70-142, Naval Surface Weapons Center, White Oak, 28 Oct 1970
26. MIL-STD-650, Method 503.1, "100°C and 120°C Vacuum Stability Test," 3 Aug 1962
27. MIL-STD-650, Method 504.1, "Reactivity Test," 3 Aug 1962

and cumbersome. Condensates are often seen in the cooler portion of the apparatus and their contribution to gas evolution depends on the vapor pressure of the liquid. Volatile reactants and products are removed from the reaction zone to the cooler parts of the apparatus. In compatibility testing samples may contain up to 50 percent of deliberately-added inert which may be grossly in excess of what the explosive will ever see in practice.

7.2.11 All of the methods summarized above have some validity, given their known limitations, but, surprisingly enough, the very simple vacuum stability apparatus persists and shows no sign of being completely replaced. Indeed, under the auspices of NATO, consideration is being given to the development of a standard vacuum stability test.

8. COMPATIBILITY TESTING PHILOSOPHY

8.1 In the standard Navy test, an explosive plus an additive are considered to be incompatible if the gas volume evolved by the mixture is greater than the sum of the gas volumes from separate tests on the explosive and additive. This standard seems somewhat arbitrary since the criteria for the vacuum stability of explosives permits the evolution of up to two milliliters of gas before the explosive is considered unstable. This situation arises because it is not known how much instability can be tolerated safely in an explosive system.

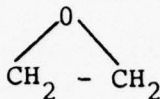
8.2 The standard stability/compatibility criteria are designed to ensure the safe storage of explosives for long time periods. Based on the storage history of standard explosives, small changes in decomposition rates may not be significant, even during prolonged storage periods of 10 or 20 years. It may not be reasonable to apply the same incompatibility criteria to a polymer designed to immobilize an explosive train for a limited time period. An unrealistic set of criteria will only increase the difficulty of finding a usable polymer composition.

9. THERMOSETTING POLYMERS

9.1 The term "polymer" refers to a large molecule made up of many simple repeating units. Network or crosslinked polymers arise when polymer chains are linked together chemically. Because of crosslinking, the polymer chains lose their mobility and the material has dimensional stability and can be considered as one giant molecule. A thermoset polymer will not melt or flow and cannot be molded. To use thermosetting resins, one must accomplish the crosslinking reaction in place. Thermoplastic polymers are not crosslinked, can usually be dissolved in some solvent, and will melt or soften and flow. It should be recognized that the above classification of polymers into two major classes is highly oversimplified.

9.2 Epoxy Resins

9.2.1 The term "epoxy" refers to a chemical group consisting of an oxygen atom bonded to two carbon atoms already united in some other way. The simplest epoxy is a three-membered ring termed α -epoxy or 1,2-epoxy. Ethylene oxide (I) is an example of this type.



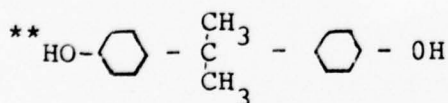
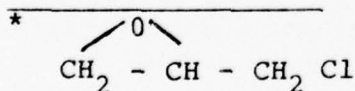
I

Ethylene Oxide

9.2.2 The three-membered epoxy ring may be referred to as an epoxide or may be called oxides such as ethylene oxide or cyclohexene oxide. Several of the more common monoepoxides have trivial names such as epichlorohydrin, glycidic acid, and glycidol. Glycidyl refers to a terminal epoxy group, the name modified by ether, ester, amine or other chemical groups. For the purposes of this report an epoxy resin is defined as any molecule containing more than one α -epoxy group capable of being converted into thermoset form. The term is also used to indicate the resins in both the thermoplastic (uncured) and thermoset (cured) states.

9.2.3 The first commercial epoxy resins were the reaction products of epichlorohydrin* and bisphenol A** to give the diglycidyl ether of bisphenol A (DGEBA). Via the epichlorohydrin route a large number of other epoxy resins are made by reaction with other compounds containing active hydrogens. These may include organic and inorganic acids, nitrogen-containing compounds and silicones. Monoepoxides other than epichlorohydrin can also be used to synthesize these resins. Another major class of epoxy resins are made by the epoxidation of olefins and from acetyl compounds (Reference 28).

9.2.4 The popularity of epoxy resins are due to a unique combination of desirable properties. These include low viscosity, good cure properties, relatively low shrinkage, excellent adhesive properties, high mechanical strength, and a high degree of versatility



28. Lee, H. and Neville, K., "Handbook of Epoxy Resins," McGraw-Hill Book Co., New York, New York, 1967

and chemical resistance. The conversion of a liquid epoxy resin to a hard thermoset solid is accomplished by the addition of a chemically-active curing agent. The conversion from liquid to solid usually does not generate any by-products.

9.2.5 There are mainly two general classes of curing agents - acids and bases. The basic curing agents are usually amines, amides, Lewis bases, and inorganic bases. Acid curing agents usually employed are Lewis acids, carboxylic acid anhydrides, organic acids and phenols. It has been shown previously that bases are incompatible with most explosives. It has been amply demonstrated experimentally that amine curing agents are not compatible with most explosives. Amine-cured epoxy resins may or may not be compatible with explosives. Some of the contributing factors are type and basicity of amine, mechanism of cure and structure of the cured resin (References 29, 30 and 31). Therefore, if epoxy resins are to be used in contact with explosives, acidic curing agents should be used. Only those acidic curing agents that appeared to be reactive enough to effect gelation and cure at room temperature and below were considered in this review.

9.3 Acidic Epoxy Curing Agents

9.3.1 Lewis Acids

9.3.1.1 These consist of compounds such as aluminum chloride, aluminum bromide, zinc chloride, boron trifluoride, silicon tetrachloride, stannic chloride, ferric chloride and titanium tetrachloride. The Lewis acids are effective catalysts for glycidyl ether resins and both linear (Reference 32) and cyclic epoxidized olefin resins (References 33 and 34). The more reactive Lewis acids give highly exothermic reactions at room temperature. For this reason they can only be used to cure small masses of resin when short pot lives can be tolerated. They should be useful curing agents at low cure temperatures.

29. Silver, I. and Anderson, H. C. "Compatibility of Epoxy-Type Plastic Materials with Explosives," NAVORD Report 4359, Naval Ordnance Laboratory, White Oak, 17 Aug 1956
30. Silver, I. "Compatibility of HBX-1, M-1, and N-5 Powders with Laminating Resins and Adhesives," NAVORD Report 3771, Naval Ordnance Laboratory, White Oak, 25 Aug 1954
31. Anderson, H. C. "The Effect of Different Amines on the Mechanism of Cure of Epoxy Resins and Explosive Compatibility," NAVORD Report 6023, Naval Ordnance Laboratory, 16 Dec 1957
32. Johnston and Greenspan, "Oxiron Resins: A Series of New Epoxy Resins," Mod. Plastics, May 1961
33. British Patent 642,800
34. U. S. Patent 2,890,209

9.3.1.2 The Lewis acids are usually used in combination with some carrier solvent. Stannic chloride may be complexed with polyols or monoethers to produce gel times at 37°C from about 1 to 5 minutes, depending on the concentration of polyol and catalyst (Reference 35). Antimony pentachloride is reported to be an effective catalyst in a triethylene glycol carrier.

9.3.2 Boron Compounds

9.3.2.1 The methylpolyborates will cure DGEBA resins providing pot lives of about 30 minutes. Some polyborates are more reactive giving pot lives of one minute at room temperature (Reference 36). Tricresyl borates react rapidly with epoxidized olefins (Reference 37). Both trimethoxyboroxine (TMOBX) (Reference 38) and tributoxyboroxine (Reference 39) will cure DGEBA at room temperature, with the former providing increased reactivity (Reference 40). TMOBX will cure many epoxides at room temperature and in some cases will cause very rapid cures with the production of rigid foams (Reference 41).

9.3.3 Metal Salts

9.3.3.1 Organic salts of polyvalent metals have been evaluated as epoxy curing agents. Stannous octoate will cure epoxy resins containing the cyclohexene oxide group at room temperature, although the cure rate is probably too low for this project. Stannous octoate may also be used as an accelerator for resins cured with polycarboxylic acids and anhydrides, polyhydric phenols, polythiols and polyisocyanates (Reference 42). Butyl titanate rapidly gels high molecular weight DGEBA resins (Reference 43). Aluminum chelates and Cr (III) tris (acetylacetonate) chelate have been used as epoxy curing agents (References 44 and 45).

9.3.4 Noncarboxylic Acids

9.3.4.1 The reaction of sulfuric and polysulfonic acids with epoxy resins to give very rapid cures has been reported

35. British Patent 882,360
36. British Patent 987,584
37. British Patent 972,952
38. British Patent 910,899
39. U. S. Patent 3,134,754
40. Haworth and Pollnow, "Boron Curing Agents for Epoxy Resins," Ind. Eng. Chem. Prod. R&D, Sep 1962
41. Lee, H. and Neville, K., "New Curing Agents for Epoxy Resins," SPEV., Mar 1960
42. U. S. Patent 3,117,099
43. U. S. Patent 2,733,222
44. Belgian Patent 623,245
45. Belgian Patent 633,621

(References 46 and 47). Liquid alkyl phosphates derived by reacting ethylene oxide with phosphoric acid provide fast, room-temperature cures with a variety of epoxy resins. Pot lives from 1 to 20 minutes have been reported (Reference 48).

9.3.5 Dimer and Trimer Fatty Acids

9.3.5.1 Dimerized and trimerized fatty acids are made by the polymerization of unsaturated fatty acids. Usually they react very sluggishly with epoxy resins unless catalyzed with amines. Research at the NAVSURFWPNCEN, White Oak Laboratory, has produced compositions consisting of epoxy resins, trimer acid and organic chromium catalysts that gel in three minutes or less at room temperature.

9.3.6 Miscellaneous Epoxy Curing Agents

9.3.6.1 A number of other classes of acidic curing agents have been used to cure epoxy resins. These include organic acids, anhydrides, polysulfides, carboxyl-terminated polyesters, aromatic-containing aliphatic acids, and substituted phthalocyanines. However, these materials react very slowly with epoxy resins and usually require elevated temperature cures. About 15 years ago, Shell Chemical Company produced an experimental, room-temperature curing epoxy adhesive which was claimed to be compatible with explosives. The adhesive did appear to be compatible with TNT based on preliminary vacuum stability tests conducted at this Center. Room temperature cure was achieved without the use of amines by the addition of triphenylphosphine (TPP) which is a good epoxy cure catalyst. The toxicity of TPP would make it a poor choice for use in a composition especially for field use.

9.4 Polyurethanes

9.4.1 The basic chemistry of the polyurethanes is that one mole of a diisocyanate reacted with one mole of a difunctional hydrogen donor will polymerize into a solid mass. No by-products are evolved unless water or a carboxyl group is present, in which case carbon dioxide gas is given off. A few of the basic reactions are:

Diisocyanate + water	= urea
Diisocyanate + hydroxyl	= urethane
Diisocyanate + amine	= disubstituted urea
Diisocyanate + urethane	= allophanate
Diisocyanate + disubstituted urea	= substituted biuret

46. U. S. Patent 2,917,491

47. U. S. Patent 2,643,243

48. Thiokol Chemical Corp., Bulletin PE 13

9.4.2 The fact that diisocyanates react readily with water would seem to rule out the use of urethanes for any application that requires polymerization in the presence of water. However, this Center has made experimental polyurethane compositions based on a low water-reactive diisocyanate (DDI*) and a hydroxy-terminated polybutadiene which cured into a solid at room temperature while immersed in water. The cured materials were soft and elastomeric and modification with short-chain polyols would be required to increase crosslink density and Shore A hardness of such systems. Polyurethane systems can be prepared from ingredients which are compatible with most explosives.

9.5 Vinyl Polymers and Unsaturated Polyesters

9.5.1 These polymers are usually crosslinked via a free radical reaction with organic peroxides the usual source of radicals. Since nitrocompounds react with free radicals and TNT is incompatible with peroxides, this type of polymer does not seem useful for the purposes of this project.

9.6 Polysiloxanes (Silicones)

9.6.1 This group of commercially-useful polymers has been used to a limited extent in some torpedo warheads. Silicone resins usually have low reactivity and must be cured at elevated temperatures. Silicone rubbers (RTV) will cure at room temperature but long gel times at low temperatures can be expected. Other problems likely to be encountered are very high viscosities at low temperatures and short shelf-life at moderate temperatures. However, many silicone systems can be devised which should be compatible with most explosives. Silicone diluents are available which will lower the viscosity of silicone polymers at low temperatures and more reactive curing agents may lower the gel time to acceptable levels.

9.7 Miscellaneous Compositions

9.7.1 Considering the difficulties involved in devising a single polymeric composition which will have all of the properties listed in Section 2.1, some consideration should be given as to whether all of these properties are absolutely necessary. It is probable that any polymeric solid injected into a fuze will immobilize its mechanical components. If some of the desired properties can be relaxed then a number of other approaches to the problem may be possible. None of the following systems have been screened for possible use in fuzes and their feasibility in such an application is unknown. They are presented for the purpose of stimulating additional approaches to the problem.

*O = C = N [D] N = C = O where D = 36 CH₂ hydrocarbon radicals

9.7.2 A catalyst could be sprayed from an aerosol container into a fuze and then followed by an injection of ethylene gas from another aerosol can. As the ethylene gas contacted the catalyst, it would polymerize into solid polyethylene. Obvious advantages are relative simplicity in field use and the ability of a gas to penetrate fine orifices that would not be filled by a viscous liquid. Such a system has been reported (Reference 49).

9.7.3 A still simpler system would consist of a thermoplastic resin dissolved in a low boiling, nonflammable solvent contained in an aerosol can. Several increments of solution would be sprayed into the fuze. After each incremental spray injection, the solvent would flash off, leaving behind a solid polymer. This idea is used commercially to package artificial snow and contact adhesives.

9.7.4 Another system could employ a molten thermoplastic or any other low melting solid. The melting would be accomplished by packaging the system in a double compartment can. Puncturing the bottom compartment would start an exothermic chemical reaction which would melt the contents of the compartment above. This idea is used commercially for self-heating food containers. There are a number of distinct advantages in the use of thermoplastic resin systems. Dependence on temperature-dependent chemical curing reactions is eliminated and thermoplastic resins, as a class, are more compatible with explosives than thermosetting polymer systems.

9.7.5 There are stiff greases made for hydraulic systems that maintain their viscosity over wide temperature ranges (~ -10 to $+30^{\circ}\text{C}$). The fuze could be pumped full of grease by the use of a hand-operated pump. It would have to be determined whether the grease was viscous enough to immobilize the fuze components.

9.8 Proprietary Polymer Compositions

9.8.1 A very large number of proprietary, two-part, ambient-temperature curing epoxy and polyurethane type adhesives, sealants and potting compounds are available commercially. Each material usually consists of two containers of equal volume and the composition is made by mixing equal weights from each container. The major component of Part A is the resin while Part B contains the curing agent. These are extremely versatile materials and are being used in a number of fields in which previous familiarity with polymer systems is lacking. Since the composition of these materials are proprietary, it is usually impossible to determine from the manufacturer's literature whether a particular material can be used in contact with explosives.

9.8.2 Epoxy Resin Systems - All two-part, proprietary, room-temperature curing epoxy resin systems are incompatible with

49. I&EC, 55 (8), 11 (1963)

aliphatic and aromatic nitro compounds (TNT, etc.), nitrate esters (NG, nitrocellulose), probably with nitramine compounds (RDX and HMX) and most of the explosives considered in this report.

9.8.3 Most of the ambient temperature epoxy curing agents are amines or modified amines and may be capable of causing serious skin and eye damage. These materials may cause, in addition, a severe rash in sensitized persons by contact with the material or its vapor. The manufacturer's toxicity information should be closely adhered to. The properly cured epoxy resin usually presents no toxic hazard but may or may not be compatible with explosives.

9.8.4 Proprietary polyurethanes may or may not be compatible with explosives. There is no way to determine compatibility without running a vacuum stability test. Some of the more volatile diisocyanate curing agents (Toluene diisocyanate) are fairly toxic. MOCA curing agent (4,4"-methylene-bis-2-chloroaniline), used until recently in a number of proprietary polyurethanes, has been identified as a potent carcinogen by OSHA. If the explosive is not very dry, diisocyanates in the polyurethane will react with the water to form CO₂ gas and eventually amines which are incompatible with most explosives.

9.8.5 There are a host of other potential problems. Even if a compatibility test indicates that a particular product is compatible with explosives, the next batch purchased may not be. Manufacturers often reformulate their products especially in today's climate of escalating ingredient costs and shortages. Some plastic materials contain liquid plasticizers which can migrate into an explosive substrate. Some diisocyanate curing agents are good solvents for TNT. Some cured polyurethanes revert to the liquid state on aging. Certainly no proprietary material should be used in contact with explosives before a compatibility test has been run.

10. CONCLUSIONS

10.1 The plastics and explosives literature have been briefly surveyed in an effort to lay a basis for the future development of a polymer system suitable for fuze immobilization requirements. Since most, if not all, Navy-approved fuze train and booster explosives are incompatible with basic materials and alkalis, it follows that a compatible system must be chemically neutral or somewhat acidic. All Navy fuze train and booster explosives, with the possible exception of lead azide, are probably compatible with weakly acidic materials at temperatures up to about 50°C.

10.2 A number of acidic thermosetting polymer systems have been presented in this report but the development of a single composition that meets all or most of the requirements listed in Section 2.1 presents a number of difficulties.

a. Viscosity - Most polymer compositions that are free-flowing liquids in the uncured state at room temperature will either become extremely viscous or solidify at temperatures as low as -10°C or -20°C . Reactive or nonreactive diluents can be added to reduce the viscosity of these systems at low temperatures. However, these additives will reduce the reactivity and increase gel times.

b. Gel Time - For every 10°C change in temperature, the rate of a chemical reaction will approximately double or be decreased by one-half. Thus, a polymer system that gels in three minutes at 25°C will not gel for a long time at -10°C . Conversely, a polymer that gels rapidly at -10°C will react violently at 25°C . By packaging the system so that variable amounts of catalyst can be added to the polymer, adjustments in the gel time as a function of temperature can be made.

c. Exotherm - The heat of the polymerization reaction, if liberated over a short time span, will result in substantial increases in the temperature of the reacting polymer. With some epoxy systems, peak exotherms may reach 250°C or higher without external heating, which is above the decomposition temperature of most explosives. As a practical matter, the peak exotherm depends on the mass of the system and the rate at which heat can be dissipated. Peak exotherms depend also on the type of polymer system and the curing agents and catalysts used. Given the high thermal capacity and conductivity of heat sinks such as metallic fuzes, in some cases high exotherms may not be a serious problem.

11. RECOMMENDATIONS

11.1 A number of polymer systems based on epoxy, polyurethane and silicone resins have been discussed which appear to meet some of the requirements in Section 2.1. No references were found which discussed the viscosity and reactivity of these materials at temperatures approaching -10 to -20°C . Apparently these resins are rarely, if ever, used at these low temperatures. Lacking this kind of data it is difficult to recommend one or two prime candidate compositions.

11.2 It is probable that a polymer system which will meet most, if not all, the stated objectives can be devised. However, prior experience indicates that this will be a fairly difficult task especially if the material must be designed for high reliability under field conditions.

11.3 If the initial objectives can be relaxed, then it is recommended that some other approaches (discussed in Section 9.7) be considered since these or similar approaches may accomplish the objectives of this task in a more simple and reliable fashion.

Appendix A

DATA ON EXPLOSIVE COMPATIBILITY

COMPAT is a data retrieval program for polymer compatibility with propellants and explosives. It is the only known central source of this type of information which was obtained from tests conducted at Picatinny Arsenal and a wide assortment of data published in the open literature. COMPAT is available to all government agencies and industry on a fee basis. For further information contact the Plastics Technical Evaluation Center, Picatinny Arsenal, Dover, New Jersey 07801.

Appendix B
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